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Final Report
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TECHNICAL REPORT

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REPORT OF INVESTIGATION OF THE RESISTANCE TO HIGH-INTENSITY THERMAL RADIATION AFFORDED BY THE APPLICATION OF WATER-SOLUBLE FLAME RETARDANTS

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Lab. Project 5046-3 Part 38
Final Report

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Technical Objective AW-7
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14 October, 1953

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Lab. Project 5046-3 Part 38
Final Report

ABSTRACT

As part of the program to develop and evaluate suitable protective measures for materials against the effects of flash fires associated with the thermal radiation of atomic explosions, a laboratory method using black filter paper media has been devised and applied in the investigation and study of several commercially available water-soluble type flame retardants. This report describes the method as well as the quantitative and qualitative modifications resulting in the combustion behavior of cellulosic material by treatment with different concentrations of each retardant. The mechanisms by which these agents are presumed to function are briefly outlined. The relative effectiveness of these products to reduce the material destruction hazards attendant upon exposure during a nuclear detonation was determined. All of the flame retardants evaluated increased the flame and glow resistance of the black paper. It may be expected that these retardants will likewise reduce the combustibility of cotton goods and other cellulosic kindling materials. The heat transfer through layers of black and white filter papers is discussed briefly.

CONTENTS

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Ref: (a) COMNYKNAVSHIPYD ltr C-S99/L5 Serial C-960-92 of 14 Mar 1950
(b) BUSHIPS restr spdltr S99-(0) (348) Ser 348-75 of 6 Apr 1950
(c) MATLAB NYKNAVSHIPYD Project 5046-3 Part 11

Encl: (1) Properties of No. 2 and No. 29 Filter Papers
(2) Classification of the Fire Retardants
(3) Critical Energy Values of Black Filter Paper
(4) Thermal Radiation Resistance of Black Filter Paper
(5) Thermal Radiation Resistance and Add-on
(6) Ignition Characteristics of the Fire Retardants
(7) Thermal Radiation Resistance of Black and White Filter Papers

AUTHORITY

1. This investigation is part of the general program proposed by reference (a) and formally approved by reference (b). The general Thermal Radiation program is under the supervision of the Armed Forces Special Weapons Project.

INTRODUCTION

2. The use of incendiary weapons in modern warfare stresses the need for protection against primary and secondary fires in defense and civilian areas. Considerable interest has been shown by both military and civilian organizations in the flameproofing of materials as an effective means of achieving increased protection against war- and peacetime fire hazards. The importance of such protection is emphasized by the increased demand, production and ever widening application of flameproofing agents. Large amounts of these agents have been required for the fireproofing of barracks, airports, industrial plants, dwellings, theatres, and other public areas. The cellulosic materials, such as wood and textiles, used in these structures and their contents and in clothing worn by civilian and military personnel, are the most combustible types and, hence, require the greatest amount of protection. This report covers the evaluation of ten of the water-soluble or temporary type of commercial flame retardants obtained from seven different manufacturers. An attempt is made to determine the extent of applicability and relative degree of material protection afforded by these agents against the specific hazard of ABD thermal radiation.

EXPERIMENTAL PROCEDURE

3. The base material selected for this investigation was a Whatman No. 29 Black Filter Paper, whose physical and chemical properties are given in enclosure (1). Strips measuring 1 X 4 inches were cut from commercial 18.5-cm-diameter stock and weighed. The specimens were immersed for one minute in a

flame retardant solution of known concentration. Pickups of 5, 10, 15, 20 and 25 per cent were obtained by using different concentrations. All solutions were kept at room temperature. The wet strips were suspended in a vertical position and allowed to air-dry for a minimum of twenty-four hours before being reweighed to determine the amount of fire retardant retained. Two of the impregnated specimens, of equal add-on, were mounted, end to end, to form an 8 inch strip, on the surface of a 1 X 8 X 3/8-inch melamine fiberglas-laminated block containing a central channel 1/2 X 8 by 1/4-in. deep. This channel or groove served as the air background for combustion. A mask, made of the same material as the block and having four U-shaped openings, covered the specimens and held them in position. With the mask in place, the exposed area was approximately 40 per cent of the total area of the strips. This assembly was secured to a carriage which moved horizontally, at a controlled speed, across a source which furnishes an irradiance of 85 cal/cm^{2}sec. Flaming and glowing characteristics were noted during and immediately after exposure. The radiated strips were weighed and relative per cent weight loss based on impregnated weight was determined for each. The specimens were examined for points of initial char formation and burn-through, e.g. cracks or other spaces through which radiation may pass freely. To each of these points was assigned a thermal energy value in cal/cm^2 and designated, respectively, as the critical energy at char-through and the critical energy at burn-through. These values were obtained by multiplying the time in seconds by the irradiance, the time being that required for the 1-cm length of strip, wherein the destructive effect occurred, to traverse the point of focus of the radiation source. The speeds employed gave radiant exposures ranging from about 1.5 to 10 cal/cm^2. A minimum of 60 strips were impregnated with each flame retardant, twelve for each of 5 add-ons. Based on the combustion behavior of the treated specimens during and immediately after exposure, the compounds were grouped as flame and glow retardants, flame retardants or non-effectives. Within each group they were classified according to the predominant chemical constituent, as determined by Manufacturers' information and qualitative tests, and are listed in enclosure (2) in order of the average difference, for the various add-ons, between the critical energies at burn-through and char-through. This difference indicates the tendency of the flameproofing agent to affect the quality and rate of formation of char resulting from the decomposition of the cellulose. The greater this difference, the more effective is the agent in promoting rapid formation and longer retention of a continuous, unbroken char barrier which may be considered opaque in thermal radiation. The relative weight loss as a function of radiant exposure was determined for each add-on, indicating quantitatively the retarding and char forming tendencies of the compounds. Combustion characterization tests of the compounds were made by carefully igniting, over a bunsen flame, a small amount of each contained in an open crucible. Observations were made of any frothing, boiling, fuming, puffing and melting. This information was used as a guide to ascertain and explain the probable mechanisms by which each compound functioned. The usual qualitative chemical procedures were employed to determine the presence of basic or acidic constituents such as ammonia, borate, phosphate, sulfite and sulfamate.

RESULTS

4. The black filter paper begins to flame at about 1.5 cal/cm^2. It chars-through and burns-through simultaneously at 4.3 cal/cm^2, accompanied by a sharp increase in weight loss. Afterglow begins at this value and is intermittent at higher energies. Afterflame was not evident at low or high energies. The relative weight loss was about 1.5 per cent/cal. between 1 and 3 cal/cm^2 and increased to about 10 per cent between 3 and 6 cal/cm^2. The flaming characteristics of flameproofed stock, up to about 3 calories, were essentially the same as those of the untreated material, except that the flames appeared somewhat shorter and more highly colored. Char-through for the treated specimens occurred at about 0.5-1 cal/cm^2 lower than that for the untreated specimens. The average burn-through critical energy for the group, for all add-ons, was 6.5 cal/cm^2, an increase of about 50 per cent over untreated stock. Specimens impregnated with 25 per cent NaCl required 5.6 cal/cm^2. The critical energies for lower pickups were the same as those for untreated paper. Afterflame was noted in a few instances for three of the compounds, for the lower add-ons. Approximately one-half of the compounds exhibited afterglow, but at higher energy densities than in the case of the corresponding untreated stock. The critical energies are listed in enclosure (3). Beyond 3 cal/cm^2 the slopes of the relative weight loss vs energy curves of flameproofed specimens, for all the add-ons, were lower than similar curves of untreated stock. At burn-through the difference was at least 50 per cent. For the non-effective NaCl-treated papers, the slopes were higher. Typical curves are presented in enclosure (4). Curves showing the affects of add-on are presented in enclosure (5). The flame retarding compounds, when heated in an open crucible gave evidence of one or more of the following stages and in the order listed; frothing and boiling with evolution of gas and vapor, fuming, puffing and swelling of the mass to many times its original volume, melting and contracting of the puffed mass into a glass-like ash. Enclosure (6) lists the behavior of each retardant. The preparation of solutions of these agents was simplified by their high solubilities in water at different concentrations. They had no "dusting" tendencies, even at high add-ons. Sodium chloride effloresced when used above 20 per cent pickup. Two of the compounds which were received as viscous solutions, appeared to "dry-out" after 3 or 4 weeks. This did not appear to affect their flame retarding properties. Qualitative tests of each agent indicated the presence of one or more of the following chemical groups: ammonium, phosphate, borate, sulfite, sulfamate, sodium and potassium. One sample appeared to have some organic phosphate as a constituent.

DISCUSSION

5. Reference (c) describes several standard procedures for the evaluation of flameproofing agents. In general, they require the exposure of a treated substance to a flame. The type of flame, its height and exposure time are all specified. Duration of time of flaming and glowing of the material immediately after flame removal, char area and other visual destructive effects are noted. In terms of these tests, information on the relative flame-and glow-retarding properties of many types of compounds is well established. Not firmly settled, however, is the knowledge of the effectiveness of these chemicals to protect materials against a high-intensity, radiant heat pulse. It was recognized that the results would be influenced by the nature and color of the base material. Hence, for laboratory study, it was decided that cellulose stock in filter-paper form satisfied most of the requirements of a suitable standard. It was adopted because of its purity, availability, combustibility, absorbability and ease of manipulation. Add-on was easily controlled. Specimen mounting presented no difficulties. Black paper had the additional advantage of possessing a relatively flat optical absorptance over a radiation band extending from 4000-10,000 A. None of the flame retardants had dusting tendencies which would appreciably modify this optical characteristic of the paper. The high burn-through value obtained for the non-effective NaCl at 85 per cent add-on may be attributed to the increased reflectance caused by efflorescence. Because of the very short time intervals involved in the thermal radiation exposure, it was difficult to determine the exact mechanisms, for each retardant, which were effective in modifying the cellulose degradation. It can be assumed, however, that the combustion theories evolved from flame exposure studies apply to short-time radiation exposures. An examination of the ignition products and behavior of each compound mixture offered some valuable clues. For example, the detection of ammonia and steam in the initial low temperature phases of ignition suggests mechanism (a) as outlined in paragraph 11 of reference (c), viz: "evaluation of non-combustible gases which envelop the material thus preventing the ignition of combustible evolution products as the material decomposes." Puffing or swelling of the fire retardant during ignition suggests the principle of "intumescence" or the formation of an insulating layer which protects the surface underneath from further thermal damage. Fusing or melting of the compound would favor the smothering mechanism or the prevention of oxygen from reaching the material. Thus, the results of the ignition behavior studies indicate that the majority of these flame retardants function by more than one mechanism. The ultimate overall visual effects of this multiple action were almost identical for each agent. Briefly, it consisted of the early formation of a charred mass whose shape and physical continuity was retained up to an average value of about 6.5 cal/cm^2. In terms of a radiation barrier, this behavior was noteworthy, although it was realized that the mechanical strength of this barrier was low. Afterglow, a more specialized combustion effect, was

caused by and appeared to be a characteristic of those retardants which in the fuming stage of ignition indicated the absence of appreciable amounts of acid vapors. These same retardants buffed considerably and had a comparatively high proportion of glass-like ignition residue similar to that left by Borax. The combustion behavior of flame-retarded white filter paper upon exposure to thermal radiation presented some interesting results. Untreated white stock of the same thickness as the black, showed almost no weight loss up to about 30 cal/cm^2. Between 30 and 35 cal/cm^2 the weight loss increased sharply to about 30 per cent at the rate of approximately 8 per cent per cal/cm^2. This was accompanied by burn-through when treated with one of the flame- and glow-proofing agents, the same 30 per cent loss in weight occurred between 20 and 30 cal/cm^2 and the rate fell to about 4 per cent per cal/cm^2. The general shape of this curve resembled that obtained with untreated black paper. Curves for black and white stock are presented in enclosure (7). Afterflame which occurred with untreated white stock was completely suppressed by flameproofing. Further, when an untreated white strip was placed over and in contact with an untreated black, the weight-loss-energy relationship of the white top layer was the same as that given by a single white layer. Up to about 15 cal/cm^2, the loss in weight of the black layer was negligible. From 15 to 35 cal, the weight loss per calorie was about one per cent. The average char-through and burn-through values of flame-retarded white filter paper for all add-ons were 13 and 28 cal/cm^2, respectively. The values for untreated stock were both about 34 cal/cm^2. This anomalous behavior of lowered resistance after flameproofing appears to be a characteristic of white stock, so that due consideration must be given this fact in a flame-retardant evaluation study. It appears, also, that observations of after-flame and after-glow behavior should be included. If these evaluation criteria of char-through, burn-through and weight loss are used and if full cognizance be given to the increased absorptance effects of early char on white material, a closer analysis of the data presented in reference (d) would indicate that those flame retardants functioned as expected. Slight scorching or charring of the Erifon, Rezgard A and Pyroset D treated cloths occurred at 1-2 calories lower than on the untreated. Complete destruction energy values of the black cotton specimens are given as 12.2 cal/cm^2. None of the flameproofed specimens was reported as completely destroyed and except for the treated white, the destruction values (not complete destruction) of the treated cloths were at least equal to those of the non-flameproofed fabrics. If this modification of the interpretation of the data of reference (d) is correct, it must be conceded that the flameproofing treatments, employing Erifon, Rezgard A and Pyroset D, are effective in retarding cloth damage.

CONCLUSIONS

6. The laboratory method of determining the radiant exposure to cause combustion effects, such as char-through, burn-through, relative weight loss, afterflame and

afterglow of impregnated black filter paper, appears valid and useful in the investigation and appraisal of water-soluble type flame retardants to reduce destruction of cellulose-like materials which are exposed to high-intensity, thermal radiation. Helpful guides in the study of combustion behavior and protective mechanisms by which flameproofing agents function, may be obtained by examination and comparison of the slopes of the weight-loss-energy-density characteristics of treated and untreated specimens and by observation of the ignition behavior of each retardant compound or mixture. Based on the results of these procedures, it may be concluded that all the commercial retardants evaluated increased the flame and glow resistance of the black paper. The chief characteristic effects were the early formation and long retention of a continuous unbroken mass of carbonaceous residue. It may be expected that these retardants will behave in the same relative order of effectiveness when applied to other similar cellulose materials, including cotton fabrics.

Approved:

H. T. Koonce

H.T. KOONCE, CAPTAIN, USN
The Director

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PROPERTIES OF NO. 2 and NO. 29 FILTER PAPERS

PROPERTY		NO. 2 U	NO. 2 R	NO. 2 S	NO. 29 U	NO. 29 R	NO. 29 S
Color			White			Black	
Thickness	Mils	8	8	8.5	9	8	8
Weight	Oz/yd^2	2.7	-	-	2.8	-	-
Reflectance (4,000-10,000A)	%	76	77	75	4.5	5.0	5.0
Transmittance (4,000-10,000A)	%	20	19.5	20	0	0	0
Absorptance (4,000-10,000A)	%	4	3.5	5	95.5	95	95
"Char-through cal/cm^2"		34	-	-	4.3	4.0	3.4
"Burn-through cal/cm^2"		34	-	-	4.3	7.0	4.3
Average Relative Weight Loss							
Range (0-3 $Calories/cm^2$)	%/cal.	-	-	-	1.5	1.1	2
" (3-6 " ")	" "	-	-	-	10	4	12
" (0-20 " ")	" "	-	-	-	-	-	-
" (20-30 " ")	" "	1	-	-	-	-	-

U = Untreated Stock
R = Rezgard "A" (10% Add-on)
S = Sodium chloride (" " ")

CLASSIFICATION OF THE FIRE RETARDANTS

GROUP I - FLAME-AND GLOW RETARDANTS

Class A - Phosphates	Manufacturer
1 - Rezgard "A"	Monsanto Chemical Co.
2 - Flameproof XWX-B	Apex Chemical Co.
3 - Akaustan	General Dyestuff Corp.
4 - Albi- "K"	Albi Mfg. Co., Inc.
5 - Flameproofing Agent 313	Glyco Products Co. Inc.
Class B - Sulfamates	
1 - Flameproof No. 290-C	Apex Chemical Co.

Group II - Flame Retardants

Class A - Borates	
1 - Quaker Diapene AB	Quaker Chemical Products Corp.
2 - Arko Flameproofing Compound AS	Arkansas Co., Inc.
3 - Arko Fire Retardant 98 B	Arkansas Co., Inc.
4 - Abopon	Glyco Products Co., Inc.

Group III - Non-Effectives

Class A - Chlorides

1 - Common Salt

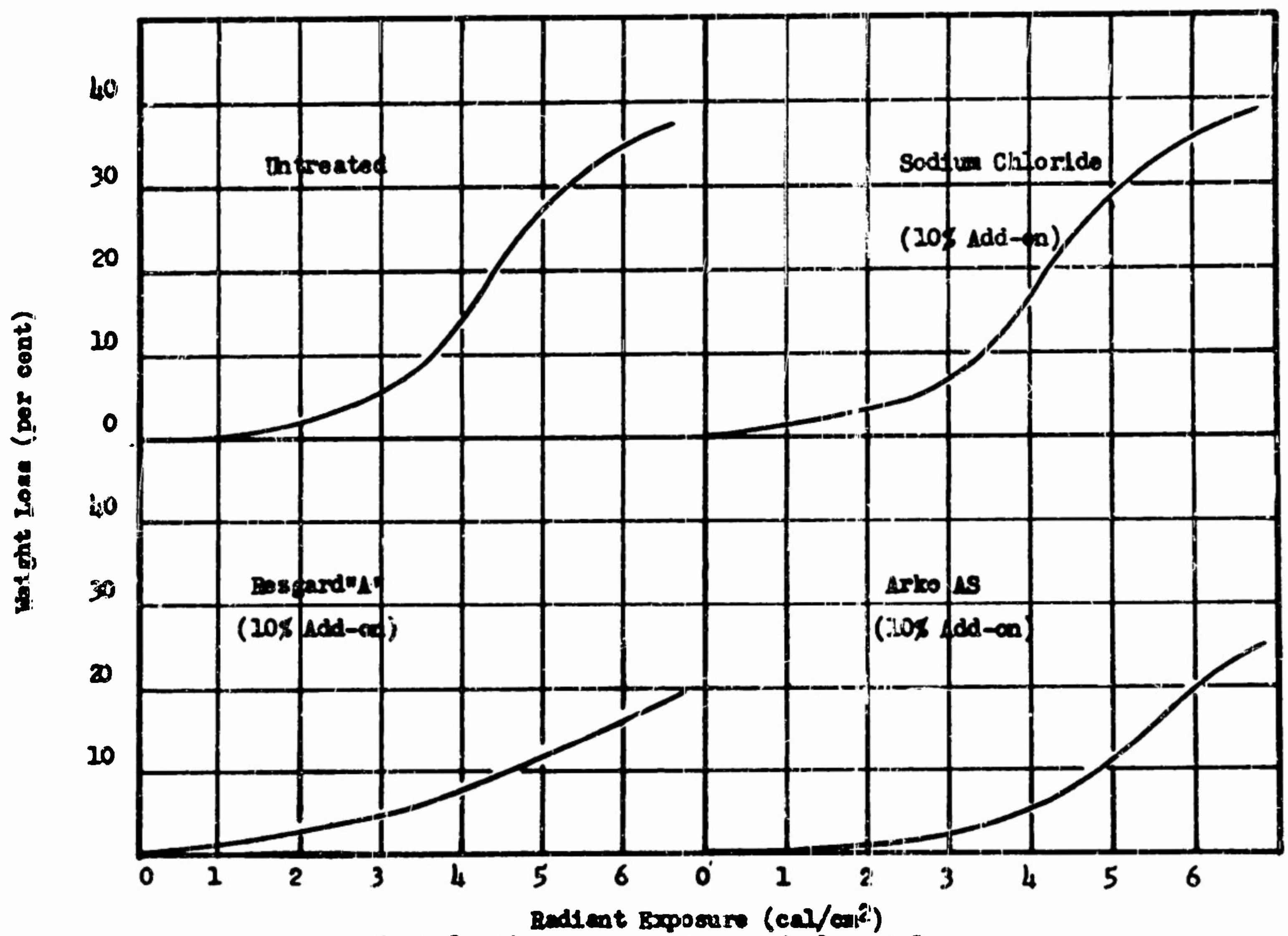
CRITICAL ENERGY VALUES OF
BLACK FILTER PAPER

Add-On Flame Retardant	5% B-T	5% C-T	10% B-T	10% C-T	15% B-T	15% C-T	20% B-T	20% C-T	25% B-T	25% C-T
Rezgard "A"	6.9	4.4	7.0	4.0	9.3	4.4	10+	4.1	10+	3.9
Apex XWX-B	7.4	4.1	6.9	4.5	7.3	4.4	6.5	3.7	6.4	3.7
Apex 290-C	5.7	4.4	6.7	4.6	6.5	5.6	-	-	7.0	5.6
Albi-"K"	6.4	4.4	6.4	3.7	6.6	4.4	6.9	4.4	6.8	3.7
Akaustan	5.7	3.5	5.9	3.7	6.6	3.5	7.3	4.1	6.4	4.4
Glyco 313	5.9	4.2	5.7	4.0	6.6	4.5	6.4	3.8	6.2	4.8
Arko 98B	5.7	4.4	5.6	4.2	5.7	4.8	5.6	4.1	5.7	4.5
Arko CPD. AS	5.6	4.1	5.6	3.7	5.6	4.4	5.3	4.0	5.4	4.2
Abopon	5.6	4.5	5.6	4.6	5.6	4.5	5.2	4.1	5.4	4.1
Diapene -AB	5.1	3.1	5.6	3.2	5.6	3.3	5.6	3.2	5.6	3.0
Common Salt	4.3	3.4	4.3	3.4	4.3	3.3	4.3	3.5	5.6	3.3

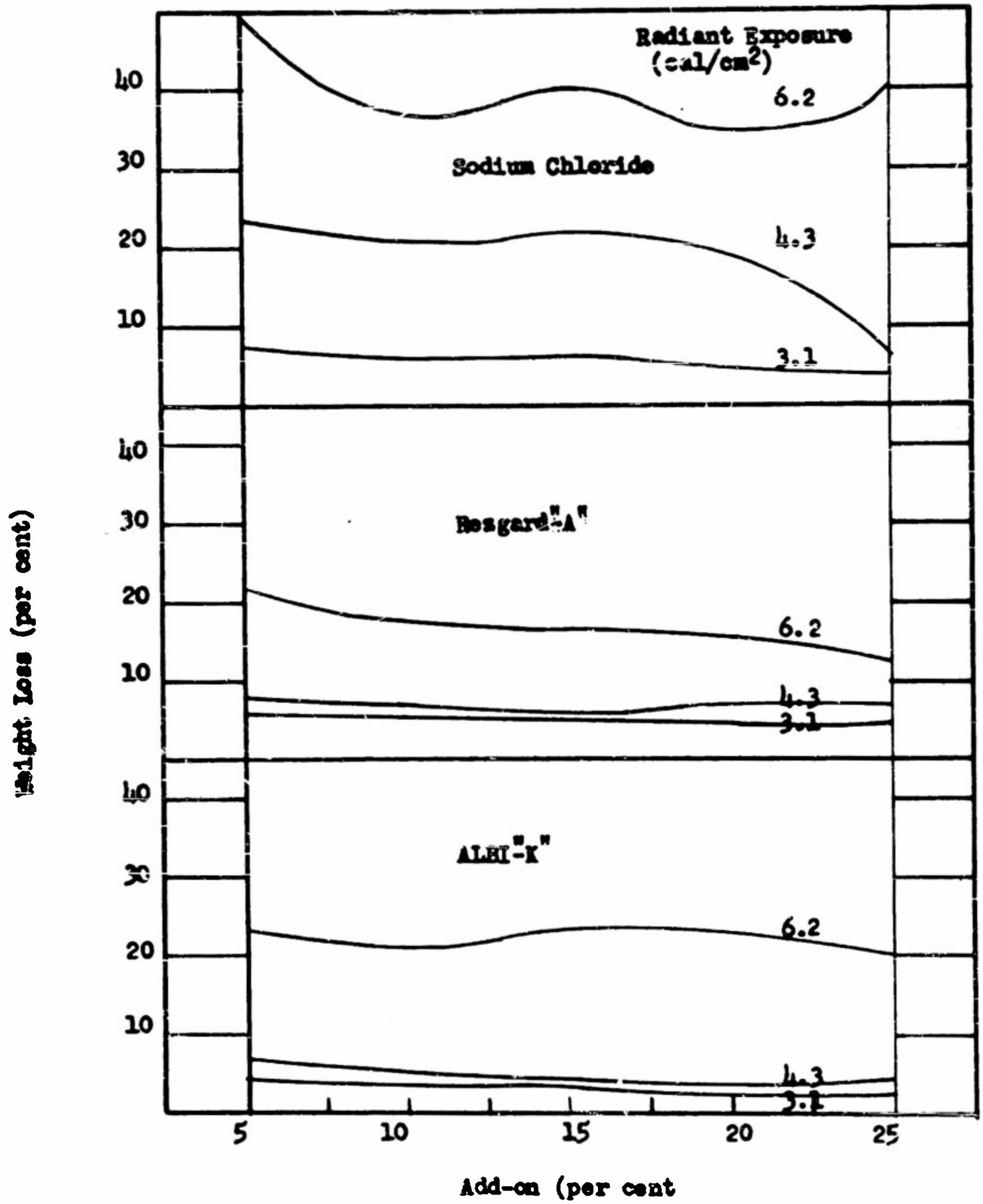
No. 29 Black Paper B-T =4.3 C-T=4.3

No. 2 White Paper B-T= 34 C-T=34

NOTES: (1) Critical Energy (Calories/Cm^2) is the minimum or critical quantity of radiant energy required to produce specific destructive effects.
(2) "Burn-Through" (B-T) that degree of destruction of material which results in the formation of a crack, hole or other opening.
(3) "Char-Through" (C-T) that degree of destruction of material which results in the initial formation of char on the back or non-radiated surface.



Thermal Radiation Resistance of Black Filter Paper
(No. 29 Whatman)

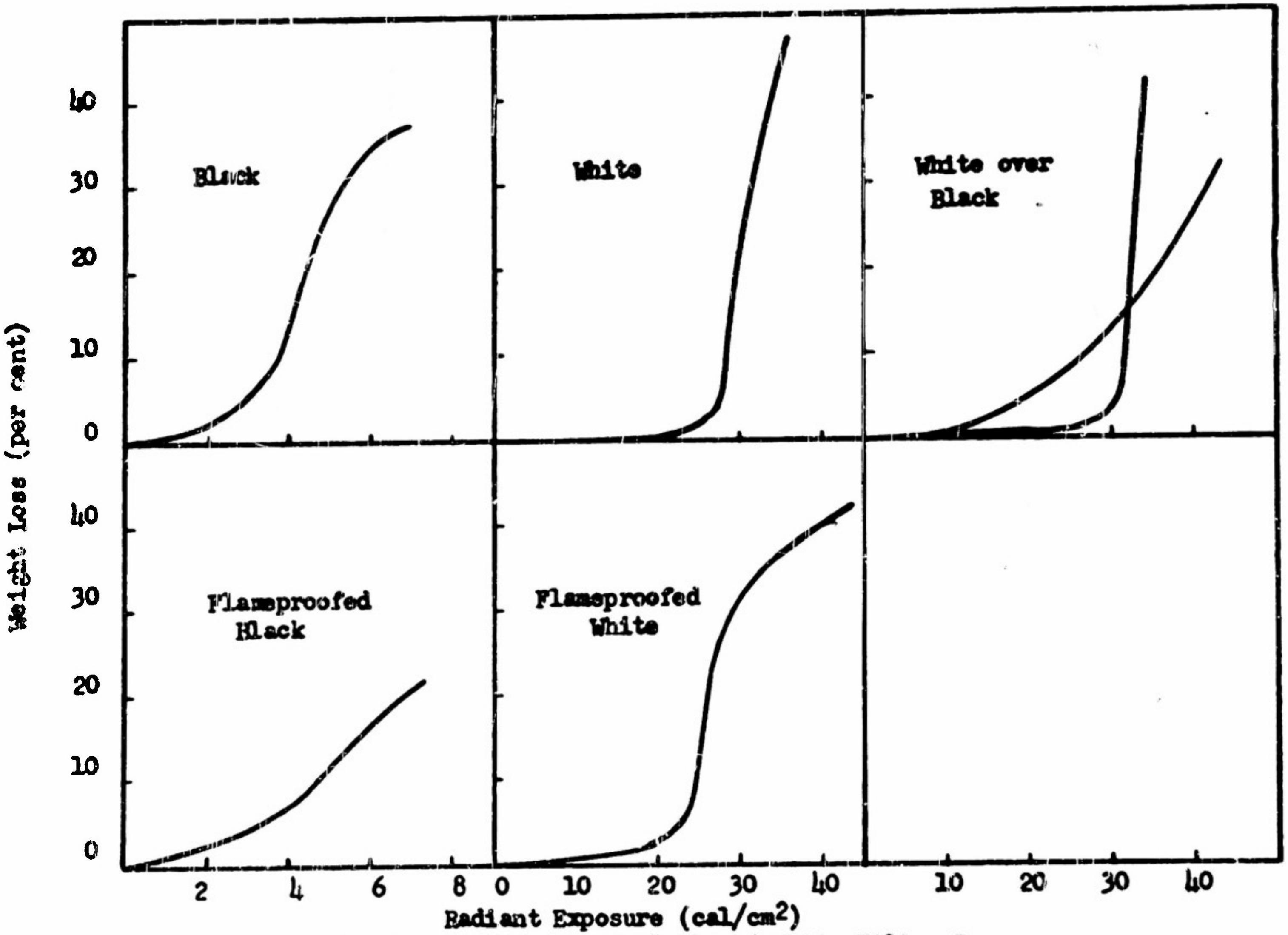


Thermal Radiation Resistance and Add-on

(No. 29 Whatman Black Filter Paper)

IGNITION CHARACTERISTICS OF THE FIRE RETARDANTS

Flame Retardant	Frothing	Boiling	Fuming	Puffing	Melting
Rezgard "A"	+	-	+	-	+
Apex XWX-B	+	-	+	-	+
Apex 290-C	+	+	+	-	-
ALBI "K"	+	-	+	-	+
Akaustan	+	-	+	-	+
Glyco 313	+	-	+	-	+
Arko 98B	+	-	+	+	+
Arko CPD. AS	+	-	+	+	+
Abopon	+	-	+	+	+
Diapene AB	+	-	+	+	+



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(No. 29 and No. 2 Whatman)

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